Enhanced Reactivity of Kraft-based Viscose-grade Dissolving Pulp by Hollander Beating Treatment

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The reactivity of rayon-grade dissolving pulp is an important parameter in the subsequent production of viscose fiber. It can determine the processability and the quality of the viscose fiber. This study focused on improving the reactivity of kraft-based rayon-grade dissolving pulp through a mechanical treatment with mechanical refining, which could induce more fiber ends and channels or reactive sites *via* fiber cutting and fibrillation. Results showed that the Fock reactivity of a softwood kraft-based dissolving pulp increased from 54.4% to 68.4%, and the viscose filterability improved from 3743 s to 11 s when the beating degree of dissolving pulp was increased from 9 ºSR to 16 ºSR. Such a treatment also led to increases in the fines content, pore diameter, water retention value, and specific surface area while decreasing the fiber length. Meanwhile, it was beneficial that the intrinsic viscosity of dissolving pulp fiber was almost unaffected by the beating treatment.

Keywords: Reactivity; Viscose-grade dissolving pulp; Beating; Fiber length; Fiber specific surface area

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INTRODUCTION

Cellulose is one of the most abundant natural materials in the world, and it has been applied to many fields due to its renewability, biodegradability, and biocompatibility (Wu *et al*. 2016; Lee *et al.* 2017). As a cellulose-rich lignocellulosic material, dissolving pulp, which is mainly produced by prehydrolysis kraft and acid sulfite processes, has attracted increased attention by virtue of its high cellulose content (90% to 99%) and promising applications in producing various cellulose derivatives, *e.g.*, viscose rayon, lyocell fiber, nanofibrillated cellulose, carboxymethylcellulose, and other biomass materials (Miao *et al*. 2015). Approximately 60% of dissolving pulp annual output is utilized for manufacturing the viscose rayon (Feng and Huang 2011).

The reactivity/accessibility of cellulose towards carbon disulfide is a critical quality parameter of dissolving pulp in the viscose rayon process (Kvarnlöf *et al*. 2006). Due to increasingly stringent environmental laws and regulations, it is imperative to use high-reactivity dissolving pulp for viscose rayon manufacturing to reduce the demand and emission of carbon disulfide. Additionally, the high reactivity of dissolving pulp can benefit viscose dope homogeneity and lower production capital. Many methods have been employed to assess the reactivity of rayon-grade dissolving pulp (Fock 1959; Christoffersson *et al*. 2002; Östberg *et al*. 2012), among which the viscose filterability and the Fock reactivity have been commonly adopted in the industry of producing dissolving pulp and academic research. The viscose filtering value, expressed in seconds (s), represents the processability of dissolving pulp in the viscose rayon manufacturing process, and the Fock reactivity, expressed as a percent (%), represents the amount of regenerated cellulose.

The reactivity of dissolving pulp is fundamentally affected by the chemical compositions, fiber structure, and fiber morphology (Engström *et al*. 2006). The compact fibrillar structure of cellulose deriving from the intra- and intermolecular hydrogen bonds especially affects reactivity along with other hydrophobic interactions that generally lead to a limited accessibility and reactivity towards reagents (Richter and Glidden 1940; Fengel and Wegener 1983). Some methods available in literature have been reported to improve the accessibility and reactivity of cellulose fibers, such as enzyme treatment (Ibarra *et al*. 2010; Miao *et al*. 2014), chemical treatment (Engström *et al*. 2006), ultrasound treatment (Tang *et al*. 2005), and mechanical treatments (Xu *et al*. 2013; Tian *et al*. 2014).

As for mechanical treatment, Papirindustriens Forskningsinstitut (PFI) refining has been used to enhance the reactivity of prehydrolysis kraft-based dissolving pulp (Tian *et al*. 2014; Miao *et al*. 2015). The PFI refining is mainly effective in fibrillating the pulp fibers rather than the fiber cutting. The refining-induced fibrillation favored the formation of inter- and intra-fibril/fiber hydrogen bonds, especially during the subsequent drying process (Bhardwaj *et al*. 2006). This would be partially responsible for the lower increased efficiency of Fock reactivity by PFI refining. It should be pointed out that so far only the Fock reactivity was assessed for the mechanical refining treatment without considering the viscose filterability. In the present study, laboratory Hollander beating, *i.e.* “Valley beater” equipment, was employed to mechanically treat the softwood kraft-based rayon-grade dissolving pulp. This beater, which works under a lower pulp consistency (1.57%), can give rise to greater cutting action compared with the main fibrillation of PFI refining. The hypothesis in this study is that the mechanical beating can cause the cutting of fibers, thereby increasing the fines content, specific surface area, and the amount of fiber ends, accessible surfaces, and channels. Consequently, this could enhance the reactivity of dissolving pulp during the subsequent xanthation step. The performance of Hollander beating was evaluated based on the Fock reactivity and viscose filterability. Other properties of dissolving pulp are also discussed, including the fiber morphological and structural changes, intrinsic viscosity, and water retention value.

EXPERIMENTAL

Materials

A softwood dissolving pulp, produced by prehydrolysis kraft-based process, was provided by Fujian Qingshan Paper Industry Co., Ltd. (Sanming, China), and its α-cellulose content was 94.0%. The pulp boards were first disintegrated by a Hollander beater (PTI Laboratory Equipment, Vorchdorf, Austria) and then were concentrated and freeze-dried. All of the chemicals used were analytical reagent grade.

Methods

Beating treatment of dissolving pulp

A 360 g dissolving pulp sample was torn into 2.5 cm × 2.5 cm flakes, and then soaked in 5 L of deionized water for 4 h. The pulp flakes were transferred into a laboratory Hollander beater after soaking, and 18 L deionized water was added to the beater to ensure a pulp consistency of 1.57%. The pulp sample was first disintegrated for 30 min before the beating treatment. Then, the beating operation was conducted based on the TAPPI T200 sp-01 (2001) method. The beating efficiency was controlled by changing the beating time (5 min, 15 min, 30 min, 45 min, and 60 min). The beating degree (°SR) was determined by employing a Schopper-Riegler beating degree tester (Qingtong & Boke Automation Technology Co., Ltd., Hangzhou, China).

Determination of Fock reactivity

The Fock reactivity of dissolving pulp samples was determined in triplicate based on an improved method (Tian *et al*. 2013).

A 0.50 g pulp sample (calculated as oven-dry pulp) was weighed and placed in a 250-mL Erlenmeyer flask with a stopper. This flask was placed in a shaker with a water bath (19 °C) after 50 mL NaOH (9% w/w) solution was added, and shaken for 10 min. The flask was sealed with the stopper as soon as 1.3 mL carbon disulfide was added, and then continued to be shook for 3 h. Subsequently, deionized water was added to the flask to give the solution a total mass of 100 g. The solution was then transferred to a tube with a stopper after being vigorously shaken. For separating the dissolved cellulose from unreacted cellulose, the solution was centrifuged at 5000 rpm for 15 min. Then, 10 mL of the supernatant was pipetted into a 100-mL conical flask, neutralized with 3 mL sulfuric acid (20% w/w), left for 15 h, and the regenerated cellulose was obtained and degassed to remove carbon disulfide. For determining the dissolved cellulose, 20 mL of sulfuric acid (68% w/w) was added to the 100-mL flask, and the suspension was shaken for 1 h to acidify the regenerated cellulose. Approximately 10 mL of potassium dichromate solution (1/6 M) was added to the flask, and the mixture was boiled for 1 h. The mixture was diluted to 100 mL when it was cooled to room temperature. Next, 40 mL of this solution was transferred into a 250-mL Erlenmeyer flask. The 40 mL solution was immediately titrated with sodium thiosulfate (0.1 N) using starch as the indicator after 5 mL potassium iodide (10% w/w) was added. The volume of consumed sodium thiosulfate was recorded to calculate the percentage of cellulose that reacted with carbon disulfide.

Determination of viscose filtering value

The viscose filtering value of dissolving pulp samples was determined in triplicate based on the FZ/T 50010.13 (2011) method.

An amount of 14.4 g of pulp sample (calculated as oven-dry pulp) and 361 mL of 13.7% NaOH solution were mixed in a 500-mL blown reagent bottle, and the suspension was fully dispersed by stirring at 3000 rpm for 5 min. Then, 11 mL of carbon disulfide was added to the suspension, and the bottle was shaken on an oscillator for 15 min. Next, the bottle was fixed on a xanthation reactor and spun (15 rpm ± 1 rpm) for 4 h. Finally, the resulting yellow solution was poured into a plastic tube with a stainless steel filter (effective filtration mesh diameter 28-mm, 10000 holes/cm^2). The filtering time from 25 mL to 50 mL and from 125 mL to 150 mL was recorded, respectively. The time difference of filtering from 25 mL to 50 mL subtracted from filtering from 125 mL to 150 mL was recorded as the viscose filtering value. The shorter of the filtering times indicated a better viscose filterability.

Measurement of fiber quality

An amount of 0.05 g of fiber (calculated as oven-dry fiber) was first dispersed by a magnetic stirrer, then the fiber length, fines content in length, and fiber coarseness were determined on a fiber quality analyzer (Morfi Compact, Techpap Inc., Grenoble, France).

Determination of fiber specific surface area and pore size

The fiber specific area and pore size of the dissolving pulp was determined based on nitrogen adsorption by using a Quantachrome volumetric gas adsorption instrument (Quantachrome Instruments Inc., Boynton Beach, FL, USA). The measurements were conducted in liquid nitrogen at a temperature of 77.35 K. The pulp samples were vacuum-dried at 100 °C for 4 h prior to the measurements.

Determination of cellulose average molecular weight

Before the molecular weight of cellulose was determined by Gel Permeation Chromatography (GPC; Waters Corporation, Milford, USA), the fiber sample was first dissolved in a lithium chloride/dimethylacetamide (LiCl/DMAc) (8%) solution. The GPC was equipped with liquid chromatography equipment (Waters 1515) together with a Waters 2414 refractive index detector. The mobile phase of the 0.5% LiCl/DMAc solution was pumped into the system at a flow rate of 1.0 mL/min. The columns used were Waters Styragel HR4 and HR5. The system was operated at a column temperature of 55 °C and the injection volume was 50 µL.

Determination of fiber water retention value

The water retention value (WRV) was determined based on the centrifugation method. A 0.5 g (oven-dry weight) pulp sample was centrifuged at 800 g force for 15 min, and then the centrifuged pulp sample was oven-dried. The WRV was then obtained from the difference of the pulp weight before and after centrifugation.

SEM observations

The fiber samples were first coated with a thin layer of gold, and then were examined on a JSM 6380 Scanning Electron Microscope (SEM; JEOL, Tokyo, Japan).

RESULTS AND DISCUSSION

Effect of Hollander Beating on Fock Reactivity and Viscose Filterability of Dissolving Pulp

As shown in Table 1, Hollander beating can dramatically improve the Fock reactivity and viscose filterability. The Fock reactivity could reach 73.85% and the viscose filtering value could decrease to 25 s at the pulp beating degree of 30 ºSR. However, with an increase in the pulp beating degree, the viscose filterability of dissolving pulp gave a poor performance when the beating degree exceeded 30 ºSR compared with the linear increase of Fock reactivity.

The increased fiber ends, surface area, and pores' exposure of fibers due to the fiber cutting might be expected to improve the reactivity. Tian *et al.* reported that the Fock reactivity of hardwood dissolving pulp could be increased by a grinding treatment with a coffee grinder under the dried condition (Tian *et al*. 2014). The Fock reactivity of the pulp sample could be increased from 49.27% to 58.32% after 6 min of grinding treatment. However, it did not investigate the effect of grinding treatment on the viscose filterability.

Table 1. Influence of Hollander Beating on Fock Reactivity and Viscose Filterability of Dissolving Pulp

Beating Degree (ºSR)	9	12.2	16	30	68	83
Fock Reactivity (%)	54.43	61.52	68.37	73.85	78.25	87.43
Viscose Filterability (s)	3743	665	11	25	7229	No pass

Figure 1 indicates the relationship between Fock reactivity and viscose filterability when the dissolving pulp was treated with a Hollander beater. As shown, the viscose filterability was not proportional to the Fock reactivity. When the Fock reactivity reached 73.8%, the time in which the viscose passed the metal net was only 25 s, but the viscose could not pass when the Fock reactivity reached 87.4%. The increase of water retention might be responsible for the latter poor performance of viscose filterability (Helmer *et al*. 2003). It also reflected that the influencing factors were different for the two different reactivity representations when the dissolving pulp was beat.

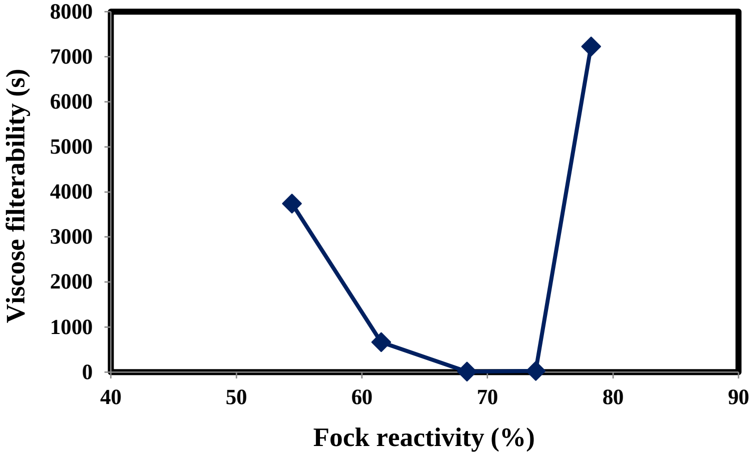

Fig. 1. The relationship between Fock reactivity and viscose filterability

Effect of Hollander Beating on Fiber Morphology of Dissolving Pulp

Table 2 shows that the beating led to an obvious decrease of fiber length and an increase in fines content. The fiber length was decreased from 1.557 mm to 1.084 mm, and the fines content increased from 11.4% to 23.4% when the beating degree of dissolving pulp was increased from 9 ºSR to 16 ºSR. This indicated that the fiber was mainly subject to the cutting action, and this behavior was further supported by the changes in fiber coarseness.

With the increase of beating degree, the fiber coarseness decreased appreciably in the beginning and then it increased when the pulp beating degree exceeded 16 ºSR. Some friction or fibrillation in the previous beating might accompany the cutting, leading to the decrease of fiber coarseness. In the later beating, the fiber was mainly subjected to cutting action that caused the increase of fiber coarseness. It was found that the coarseness of wheat straw pulp treated with PFI mill decreased faster than that of the pulp treated with a Hollander beater (Liu *et al*. 2008).

Table 2. Influence of Hollander Beating on Fiber Length, Fines Content, and Fiber Coarseness of Dissolving Pulp

Beating Degree (ºSR)	9	12.2	16	30	68	83
Fiber Length (mm)	1.557	1.393	1.084	0.75	0.551	0.432
Fines Content in Length (%)	11.4	14.9	23.4	45.8	61.9	71.8
Fiber Coarseness (mg·m^{-1})	0.0567	0.0566	0.0525	0.0545	0.0567	0.0574

The fiber morphology shown in Fig. 2 further evidenced the cutting behavior. More short fibers and fiber ends were observed in Figs. 2(c) and 2(d) due to the beating compared with the control samples as shown in Figs. 2(a) and 2(b). In addition, more pores appeared on the fibers. Many end pores in the fibers shown in the Figs. 2(c) and 2(d) could provide more access channels for the reagents (sodium hydroxide and carbon disulfide), and the reactivity of dissolving pulp was consequently improved. When the bleached soft kraft pulp fiber was mechanically treated by the Hollander beater, more banding, thick fibers, and fines were produced, indicating that the fiber was mainly cut rather than fibrillated (Fang *et al*. 2010).

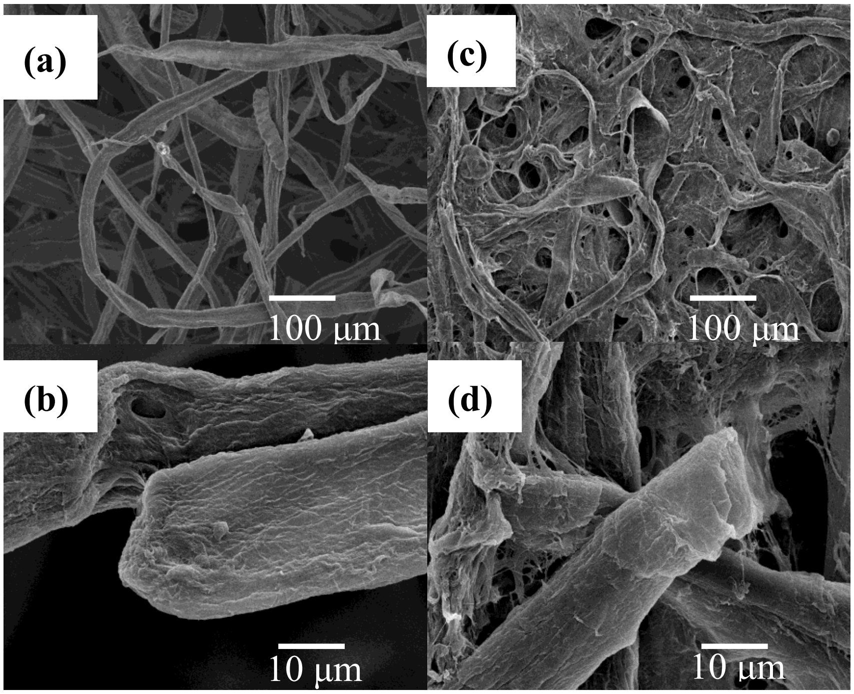

Fig. 2. The SEM of control pulp samples: (a) and (b) 9 ºSR at 200×, and beat pulp samples (c) and (d) 30 ºSR at 2000×

Effect of Hollander Beating on Fiber Specific Surface Area, Pore Size, and Water Retention Value of Dissolving Pulp

The increased fines and the newly exposed fiber ends and pores could contribute to the increases in the fiber surface area and thus the accessibility of cellulose fibers (Mou *et al*. 2013). Table 3 shows that the beating caused an increase in both fiber specific surface area and pore diameter. The increase of fiber specific surface area was beneficial for the adsorption of reagents on the fiber, and the increase of pore diameter facilitated the penetration of reagents to the reaction sites beyond the exposed fiber surfaces. Both increases consequently contributed to the improvement of dissolving pulp reactivity.

Table 3. Effect of Hollander Beating on Fiber Specific Surface Area, Pore Diameter, and Water Retention Value

Beating Degree (ºSR)	9	30
Specific Surface Area ($m^2 \cdot g^{-1}$)	2.20	2.74
Pore Diameter (nm)	3.825	4.318
Water Retention Value (%)	81.36	154.54

The WRV is mainly used to characterize the degree of fiber swelling and hydration, which is a key quality in textile fibers. Table 3 shows that the WRV of dissolving pulp fiber increased dramatically with Hollander beating. The suitable increase of WRV could promote the alkaline swelling in the additional alkali steeping step. Liu *et al.* (2007) discovered that the WRV and specific surface area increased with the increase of beating degree of bleached eucalyptus kraft pulp fiber after being refined by a PFI refiner. When the pulp beating degree increased from 19.0 ºSR to 72.5 ºSR, the WRV increased from 152.1% to 261.7%, and the specific surface area increased from 2.78 $m^2 \cdot g^{-1}$ to 6.58 $m^2 \cdot g^{-1}$.

Furthermore, the increase of WRV was mainly induced by the appearance of more ends and pores of fibers. It is worth noting that the pores not only in fibers, but also on the surface of fiber microstructures, contributed to the increase of WRV (Bosen 1970). However, the viscose filterability was negatively affected when the water retention value was too high. Therefore, the beating degree of dissolving pulp should be kept at a reasonable level to obtain an increased Fock reactivity and viscose filterability.

Effect of Hollander Beating on Intrinsic Viscosity and Molecular Weight of Dissolving Pulp

The results on intrinsic viscosity and average molecular weight of cellulose are shown in Table 4. Although the beating degree was increased dramatically from 9 ºSR to 83 ºSR, the intrinsic viscosity and average molecular weight declined only a minor degree. A viscosity reduction of 7.3 mL/g and a molecular weight reduction of 1835 was indicated for the pulp sample with a beating degree of 83 ºSR. These reductions indicated that the Hollander beating had a slight effect on the cellulose intrastructure. It must be stressed that the constant average molecular weight of cellulose with a higher reactivity was more favorable for the processing of dissolving pulp, production of viscose fiber, and the strength of viscose fiber.

Table 4. Influence of Hollander Beating on Molecular Weight

Beating Degree (ºSR)	9	16	83
Intrinsic Viscosity ($mL \cdot g^{-1}$)	362.2	362.6	354.9
Average Molecular Weight	271635	271442	269800

CONCLUSIONS

1. A mechanical treatment employing Hollander beating was utilized to treat softwood kraft-based viscose-grade dissolving pulp to improve its reactivity. The mechanical beating resulted in increased fines content, pore diameter, and fiber specific surface area, which all contributed to the improvement of Fock reactivity and viscose filterability. The beating degree of 16 ºSR could be considered as the optimized beating process based on the reactivity property of dissolving pulp.
2. The increase in WRV of dissolving pulp, due to Hollander beating, might have been partially responsible for the later decline of viscose filterability.
3. Compared with the untreated pulp sample, the viscosity and the average molecular weight were slightly decreased.
4. The application of the Hollander beating treatment to softwood kraft-based dissolving pulp can provide a simple and effective method for improving its performance in the viscose rayon manufacturing process.

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